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REPORT

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STUDIES OF THE EFFECTS OF SUB-ZERO STORAGE TEMPERATURES
ON THE PROPERTIES OF PRE-MIXED POLYSULPHIDE SEALANTS

John W. Barber, Peter J. Hanhela,
Robert H.E. Huang and D. Brenton Paul

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ABSTRACT

Pre-mixed polysulphide sealants were stored at -20, -30 and -40°C for selected intervals and then tested for conformity to the requirements of the appropriate MIL-specification in regard to cure rate or hardness, application time, tack-free time and peel strength. For Pro-Seal 899 and PR-1750 sealants, no significant change in hardness or peel strength occurred after prolonged storage at temperatures of -20°C and below; a minor decline in peel strength occurred with PR-1422. Application properties were also maintained for extended periods and at -40°C tests terminated only due to consumption of samples after storage times ranging from 7 to 16 weeks. These storage lives far exceed manufacturers' limits. Batch variations and stricter observation of class requirements could influence recommended storage times but at -40°C even materials such as PR-1750, which have a close tolerance in application life rating, may be pre-mixed and be safely stored for at least 10 weeks. Both class A and B sealants may be stored in pre-mixed form provided their application lives are at least 2 hours. Thawing is most conveniently and efficiently achieved by allowing the frozen sealant to equilibrate at room temperature.

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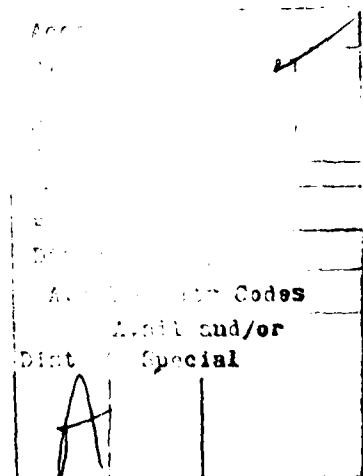
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Premixed polysulphide sealants were stored at -20, -30 and -40°C for selected intervals and then tested for conformity to the requirements of the appropriate MIL-specification in regard to cure rate or hardness, application time, tack free-time and peel strength. For Pro-Seal 899 and PR-1750 sealants, no significant change in hardness or peel strength occurred after prolonged storage at temperatures of -20°C and below; a minor decline in peel strength occurred with PR-1422. Application properties were also maintained for extended periods and at -40°C tests terminated only due to consumption of samples after storage times ranging from 7 to 16 weeks. These storage lives far exceed manufacturers' limits. Batch variations and stricter observation of class requirements could influence recommended storage times but at -40°C even materials such as PR-1750, which have a close tolerance in application life rating, may be premixed and be safely stored for at least 10 weeks. Both class A and B sealants may be stored in premixed form provided their application lives are at least 2 hours. Thawing is most conveniently and efficiently achieved by allowing the frozen sealant to equilibrate at room temperature.

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STUDIES OF THE EFFECTS OF SUB-ZERO STORAGE TEMPERATURES ON THE PROPERTIES OF PRE-MIXED POLYSULPHIDE SEALANTS

1. INTRODUCTION

Polysulphide sealants are used extensively in modern aircraft for sealing integral fuel tanks, crew modules, canopies and water tanks. They are also employed as form-in-place gaskets, and as aerodynamic smoothing materials on the external surfaces of aircraft. Sealants suitable for use in these roles are required by RAAF to meet the conditions of two specifications. For aircraft such as the Mirage 111, P-3 (Orion), C-130 (Hercules) and the Canberra, the sealants must conform to MIL-S-8802 [1] which requires that they maintain performance to temperatures up to 121°C (250°F). The most commonly used approved material is PR-1422, the only military sealant manufactured in Australia and which is produced by Selleys Chemical Company under licence from Products Research and Chemical Corporation (PRC) of the USA. For the F-111C, however, the increased aerodynamic heating which results from higher speed flight necessitates the use of a sealant qualified to MIL-S-83430 [2]. This specification stipulates that the sealant must retain its integrity up to 121°C (250°F) under continuous operating conditions and to 182°C (360°F) for intermittent, short periods. The two sealants which originally were qualified to MIL-S-83430 were Pro-Seal 899 and PR-1750, both of which have had to be imported from the USA. Sealant repairs to Australian F-111C aircraft have almost exclusively involved Pro-Seal 899. As of 15 May, 1980, however, the Air Force Materials Laboratory, USA advised that all classes of Pro-Seal 899 had been deleted from the Qualified Products List for MIL-S-83430.

Aircraft sealants frequently need to be replaced because of fuel or air leaks or as a consequence of rework or replacement of sealed components. After the service life of fuel tank sealants is exceeded, it is often necessary to undertake an entire desealing and resealing programme [3,4]. Whereas small sealing repairs may be accomplished by hand mixing of limited quantities of sealant, for major repairs (e.g. F-111C E service) and resealing operations, the most convenient method is to mechanically pre-mix a large

batch of sealant which is immediately packaged in a suitable plastic cartridge and snap frozen. The cartridges are then stored at low temperatures (-20 to -40°C) until required. Thirty minutes equilibration at ambient temperature is sufficient to thaw the frozen sealant. Apart from the reduction in mixing time this procedure has the added advantage that quality control tests need be carried out on only the one large batch rather than numerous small mixes.

At sub-zero temperatures the sealant curing reaction still continues, but at a much slower rate than under ambient conditions. In addition, fillers or other additives may concentrate or settle out under storage. The mixed sealant therefore slowly changes character over its storage life. Knowledge of safe storage limits of pre-mixed sealants is therefore essential for planning of refurbishment programmes. Little data is available on this subject, however, and much of this information is of a general nature and does not relate to specific sealants.

The Thiokol Chemical Corporation supplies the liquid polymer for nearly all polysulphide sealant manufacturers. The effect of storage was examined by Thiokol for four 'typical sealant formulations' which were held at temperatures ranging from -40°C (-40°F) to 49°C (120°F) after mixing [5]. The results are depicted in Figure 1. For these sealants of unrevealed composition, the storage life at -40°C was found to be in excess of three months.

No information on the effect of low temperature storage is provided for Coast Pro-Seal products. Products Research Corporation data sheets, however, indicate that the application life of polysulphide sealants doubles for every 6°C (10°F) drop in temperature, is reduced by 45 minutes as a result of freezing and thawing operations and that high humidity also attenuates application life [6]. Additional details are provided for PR-1422 in Table 1.

Neither the MIL-specifications nor the related General Dynamics FMS-1044 specification [7] demand that sealants meet refrigerated storage requirements. MIL-S-83430, however, recognises that it is acceptable to snap freeze sealant of classes B-2 to B-6 prior to carrying out particular tests. This procedure is described but the maximum allowable storage times are set conservatively at only four days.

US Air Force documents also emphasise the information shortfall. For example, a Process Order from Sacramento Air Logistics Center at McClellan Air Force Base states that there are no established directives for control, storage and determination of the end of the application life of snap frozen pre-mixed sealant [8]. An apparently arbitrary arrangement exists which requires that sealants stored in excess of 21 days at -40°C are discarded. In practice the volume of sealant used on the base is sufficiently large that the necessity to discard material rarely arises. For RAAF bases, however, relatively smaller usage and difficulties associated with overseas procurement of limited shelf life items demand that reliable data on storage properties be obtained.

In this report the effects of storage at various time intervals and temperatures on the performance of three pre-mixed polysulphide sealants, PR-1750, PR-1422 and Pro-Seal 899, are discussed. The effect on critical sealant properties was used to monitor the useful lifetime of the mixed sealants.

2. EXPERIMENTAL

2.1 Materials

The sealants under investigation were PR-1750 B-2 and B-6, Coast Pro-Seal 899 B-2, B-6 and A-2 (qualified to MIL-S-83430) and PR-1422 B-2 (qualified to MIL-S-8802D).

2.2 Mixing and Conditioning of Sealants

Sealants were mixed according to manufacturers' recommendations in a Semco pressure mixer model S-1350 in order to ensure sample uniformity, minimise entrapment of air and allow direct charging into 70 g (2.5 oz) polyethylene cartridges. Control samples were taken directly after mixing and tested (see below) to determine compliance with the relevant specification and provide a guide to the degree of tolerance of the batch within the specification. The filled cartridges were immediately encased in polyethylene bags, snap frozen by immersion in a dry-ice ethanol bath for 30 min and then refrigerated at controlled temperatures of $-20^{\circ} \pm 2^{\circ}\text{C}$, $-30^{\circ} \pm 2^{\circ}\text{C}$ and $-40^{\circ} \pm 2^{\circ}\text{C}$.

After various periods of cold storage the frozen cartridges were removed and thawed by two procedures. The first method required the cartridges to equilibrate at room temperature for 30 min. Zero time was taken as the time of removal of the cartridge from cold storage. The second method, as described in MIL-S-83430, required the frozen cartridges to be stabilised at $-55^{\circ} \pm 1^{\circ}\text{C}$ for 2 hours. Thawout was accomplished by immersion of the frozen cartridges, still encased in the polyethylene bags, in a water bath at $49^{\circ} \pm 1^{\circ}\text{C}$ for 18 min. The end of the 18 min period was taken as time zero.

2.3 Test Methods

For each type and class of sealant at a given temperature, duplicate cartridges were withdrawn at appropriate intervals, thawed by the alternative methods and subjected to the following tests :

2.3.1 Application Time (rate of extrusion in grams/minute)

Using a Semco model 250 sealant gun with a constant air pressure of 90 ± 5 psig, the sealant was extruded through a nozzle with an orifice diameter of 3.2 ± 0.13 mm into a tared receptacle. The amount of sealant extruded per minute was then recorded at half to one hourly intervals.

2.3.2 Tack-Free Time

Aluminium panels ($1 \times 70 \times 152$ mm) conforming to temper T6 of specification QQ-A-250/13 were cleaned with a MIL-C-38736 cleaner and inserted into polytetrafluoroethylene (PTFE) coated metal frames of suitable dimensions to form moulds. Sealants were then cast to a depth of 3.2 ± 0.4 mm for class B and 1 ± 0.25 mm for class A materials.

When cured at $25^\circ \pm 1^\circ\text{C}$ and $50 \pm 5\%$ relative humidity to the specified tack-free time, the sealants were tested for their tack-free properties. Two 25×152 mm strips of polyethylene film 0.1 ± 0.05 mm thick were applied to the sealant using light finger pressure. Two minutes after applying the film, the strips were slowly and evenly withdrawn at right angles to the sealing compound surface. Adherence of sealant to the film indicated sealant failure.

2.3.3 Standard Cure Rate (Hardness)

Sealants were cast into PTFE coated rectangular metallic moulds ($70 \times 152 \times 3.2$ mm). The class A sealant was made up by three equivalent applications, allowing 2 hours between each to permit release of solvent. Class B sealant, however, required only one layer. When cured at $25^\circ \pm 1^\circ\text{C}$ and $50 \pm 5\%$ relative humidity to the specified time, hardness readings (average of 10) were taken on a doubled back-to-back 3.2 mm thick specimen. To maintain consistent results, the Shore maximum reading durometer attached to a Conveloader was used. A hardness reading of less than 35 indicated sealant failure.

2.3.4 Peel Strength

Aluminium panels (QQ-A-250/13, T6) measuring $1 \times 70 \times 152$ mm and coated with alodine (MIL-C-5541A) and corrosion preventing polyurethane (MIL-C-27725B) were inserted into PTFE coated metal frames of suitable dimensions to form moulds. Sealants were then cast to a thickness of 3.2 mm. Over each moulded sealant was placed a 70×305 mm strip of flat, degreased aluminium wire (20-40 mesh). An additional layer of sealant, 0.8 mm thick, was applied over the wire mesh to ensure complete impregnation. The test panels were cured at $25^\circ \pm 1^\circ\text{C}$ and $50 \pm 5\%$ relative humidity for 48 hours and a further 24 hours at 60°C . At the completion of the cure the panels were stabilised for 2 hours at 25°C prior to peel tests. A 25 mm wide section of the wire mesh and sealant on the panel was stripped back at an angle of 180 degrees to the metal panel in a tensile machine (Instron model 1026) at a jaw separation rate of 50 mm per minute. To promote adhesive failure during testing, three cuts at 25 mm intervals were made through the sealing compound to the panel. Results obtained were numerical averages of the peak loads; failures of the sealing compound to the wire mesh were not included.

3. RESULTS AND DISCUSSION

Most domestic freezers operate at approximately -17°C and if equipped with a fast freeze cycle can achieve -30°C for short periods. The freezing compartments of two-door refrigerators can be held at -25°C and commercial

freezers are available which can maintain -40°C on continuous operation. For the purposes of this survey, storage temperatures of -20°C, -30°C and 40°C were selected in order to cover the range of options available from readily purchased refrigeration systems. The acceptability of pre-mixed sealant under storage was followed by monitoring critical performance and application properties as a function of storage time. The tests which were selected were tack-free time, application life, cure rate and peel strength.

Individual components of two-part polysulphide sealants undergo chemical changes when stored at room temperature and as a consequence many sealants can be guaranteed to remain within specification for only a limited period, usually 6-9 months. It is clear from Figures 2-5, however, that low temperature storage of pre-mixed sealants suppresses the rate of such reactions to a minimal level since there is little change in properties such as hardness, cure rate or peel strength even after 15 weeks storage at -20° to -40°C. In conjunction with the application life results, it is also evident that very little reaction has occurred between the base polymer and the curing agent (oxidant) at temperatures of -20°C or lower. In only one case, that of PR-1422 B-2, was there any significant deterioration in the performance and this showed as a slow decline in Shore hardness. Although the same trend persisted for all three storage temperatures, the hardness never fell below specification requirements. Measurements of peel strength showed a wider scatter than hardness but little alteration in performance was evident with storage.

Since performance properties are unimpaired after low temperature storage, the only constraints are those relating to application and handling. The periods for which pre-mixed sealants may be stored and still meet application time and tack-free requirements are depicted in Figures 6-11 and summarised in Table 2. In Figures 6a-6e the term 'application life' refers to the period during which the sealant is able to meet an extrusion rate of 15 g/min. The performance of the sealants in many cases exceeded expectation and insufficient material was available to determine the time to ultimate failure. In these instances, however, the established trend was adequate to indicate storage reliability.

The simplest and most effective thawing procedure is to allow the sealant to equilibrate at room temperature for 30 min after withdrawal from storage and the results in Table 2 were obtained by this method. Thawing was also carried out by the method described [2] in MIL-S-83430 which requires that the frozen cartridge first be stabilised at -55°C for 2 hours and then immersed at 49°C for 18 min. This procedure accelerates curing compared with room temperature equilibration as considerably attenuated storage times result (Figures 6,7). The process clearly is not intended as a practical thawing procedure and is designed to provide uniform conditions for sealants under test. At McClellan Air Force Base in California, where the volume usage of sealant is very high, thawing is achieved by placing the frozen cartridges in a microwave oven for 3 min but this method was not examined in the present study.

Zero times for room temperature thawing procedures were taken as the time of removal from refrigerated storage. The results for application life are therefore conservative since 15-30 min are required for the frozen sealant to approach the consistency of freshly mixed material. As thawing

and curing proceed concurrently, however, the condition of the pre-mixed sealant can never duplicate that of a fresh mix and a uniform zero time was preferred rather than an estimate which would change for each storage temperature. This reduction in application life due to the simultaneous thawing and curing processes becomes quite significant in the case of B-2 sealants. The fast curing A- $\frac{1}{2}$ and B- $\frac{1}{2}$ sealants are not suited for pre-mixed storage because of this effect.

It is clear that low temperature storage of mixed sealants can be practically exploited, especially at temperatures of -30°C and below, without detriment to the properties of the sealant. In addition, the storage life of such sealants is considerably extended and at -40°C far exceeds those limits suggested by manufacturers. The actual storage life, however, will be subject to manufacturing consistency between batches of sealant. The Coast Pro-Seal sealants, for example, generally pass the application life test by a large margin and B-2 grades often perform comparably with B-4 (Figures 8,9). The Products Research sealants, however, are tailored more closely to the class ranges and there is little margin of latitude (Figures 8,9).

Any change in policy by sealant manufacturers, however, which results in the production of sealants with closer tolerances in application life could affect specific conclusions reached in this study. Hence, it is more instructive to consider the effect of low temperature storage on application life in terms of changes in actual extrusion rate measurements with time rather than the simple pass/fail specification criterion. This is depicted in Figures 8 and 9 from which it can be seen that at temperatures of -30°C and below the change in extrusion rate is very small. Consequently, it may be predicted that even for sealants which comply with specifications with little margin of latitude (e.g. PR-1750), satisfactory storage can be expected over long periods.

A limited examination of Pro-Seal 899 A-2 sealant showed that low temperature storage of the mixed sealant could be achieved satisfactorily (Table 3). No significant change in properties occurred between freshly mixed and refrigerated materials and it is concluded that no separation of solvent or settling of components took place. After 20 days storage the -20°C samples failed the application life test (1.25 h). The cartridges held at -40°C varied only marginally over the test period with viscosities at 2 hours of 705, 695 and 820 poises after 6, 14 and 20 days storage respectively. For practical convenience it is suggested that pre-mixed A class sealants should be stored in a can or carton rather than a cartridge to allow free access of a brush after thawing.

4. CONCLUSIONS

1. The rate of cure, hardness and peel strength of pre-mixed polysulphide sealants are not significantly impaired after many weeks storage at sub-zero temperatures. The only restriction to the use of such materials is the ability to extrude the sealant on to a surface.
2. Both class A and B sealants may be stored in this fashion provided they have application lives of at least 2 hours.

3. Thawing is most conveniently and efficiently achieved by allowing the frozen cartridges to equilibrate at room temperature.

4. Application life requirements are still complied with after low temperature storage for many weeks. Storage at -20°C is not recommended if it is feasible to obtain refrigeration to maintain -40°C since the extended safe life at the lower temperature is considerable. Some grades of sealant were stored in excess of 16 weeks without approaching application life specification limits. All studies at -40°C terminated due to lack of samples rather than failure of the sealants. Safe limits are listed in Table 2.

5. Batch variations and stricter observation of class requirements could influence recommended storage lives. Nevertheless, at -40°C even sealants such as PR-1750, which are manufactured with a close tolerance in application life rating, may be safely stored for at least 10 weeks.

5. REFERENCES

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TABLE 1

MANUFACTURER'S RECOMMENDED STORAGE LIVES FOR PRE-MIXED
PR-1422 SEALANTS

Temperature (°C)	Storage Life (Days)	
	*Class A-2 and Above	*Class B-2 and Above
-29	3	15
-40	7	30

*Class A sealants are brush applied, Class B are applied by extrusion gun or spatula.

TABLE 2

LABORATORY DETERMINED STORAGE LIVES OF PRE-MIXED POLYSULPHIDE
SEALANTS AT VARIOUS TEMPERATURES

Sealant	Storage Life (Days)		
	-20°C	-30°C	-40°C
PR-1422 B-2	11	>47	>70
Pro-Seal 899 B-2	28	>>110	>>110
Pro-Seal 899 B-6	>100	>>100	>>100
PR-1750 B-2	13	35	>>47
PR-1750 B-6	7	21	>50
PR-1422 A-2	17	>20	>>20

TABLE 3
VARIATION IN PROPERTIES OF PRE-MIXED PRO-SEAL 899 A-2 SEALANT ON STORAGE
AT -20, -30 AND -40°C

Test*	6 Days				14 Days				20 Days			
	-20°C	-30°C	-40°C	-20°C	-30°C	-40°C	-20°C	-30°C	-40°C	-30°C	-40°C	
Peel Strength: lb/in width N/m width	28 4900	31 5400	31 5400	22 3800	28 4900	29 5100	38 6600	38 6600	43 7500	41 7200		
Cure Rate (Shore hardness)	40	42	40	38	36	36	37	37	37	37	36	
Tack-Free Time	Pass	Pass										
Application Life (viscosity after 2h, in poise)	1120	852	705	1490	912	695	3040	1340	820			

*MIL-S-83430 Requirements: Peel Strength - 20 lb (min); Cure Rate - 35;
 Application Life - 2500 poise (max).

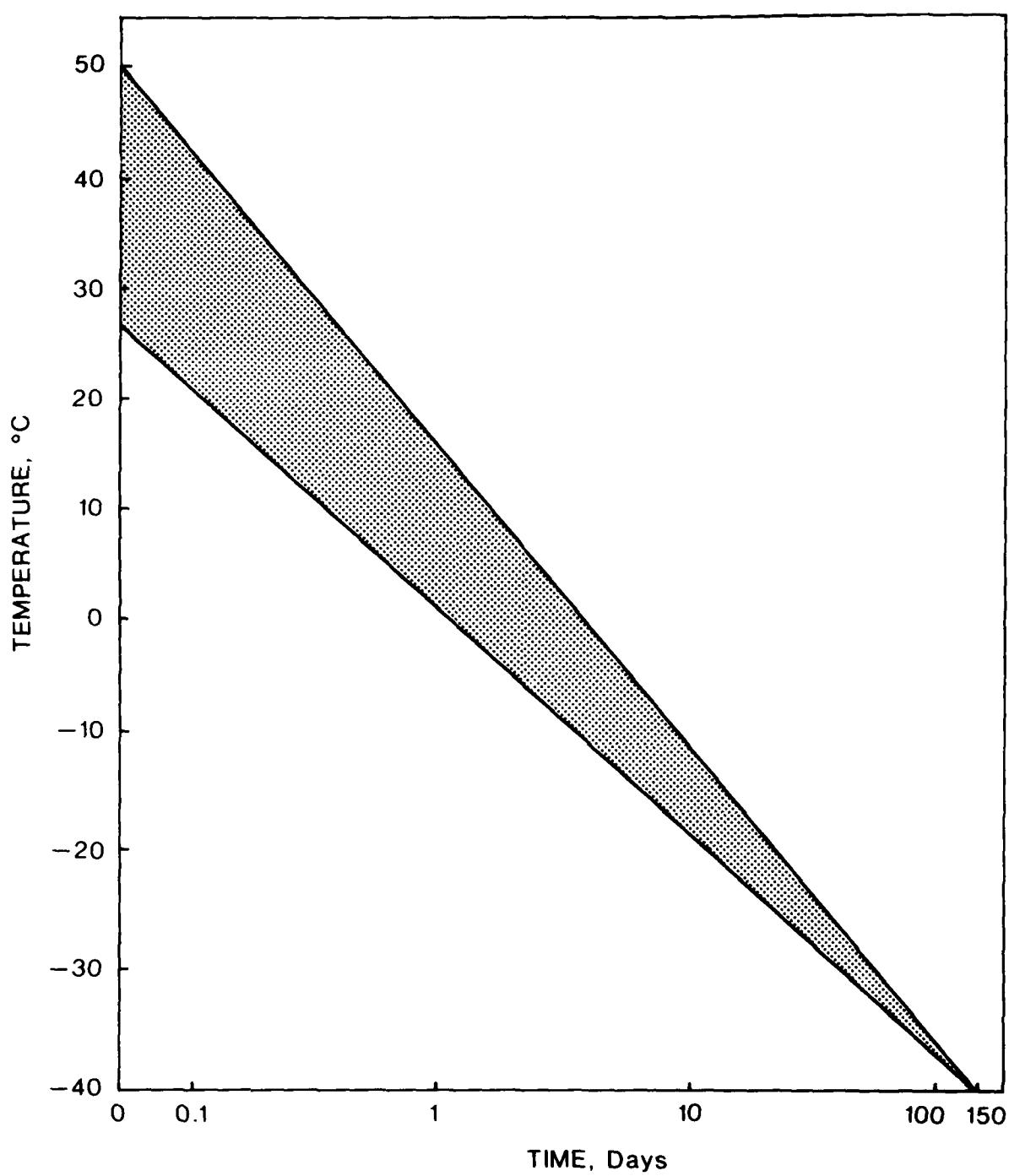


FIG. 1 - Effect of low temperature on storage life of polysulphide sealants (thiokol data).

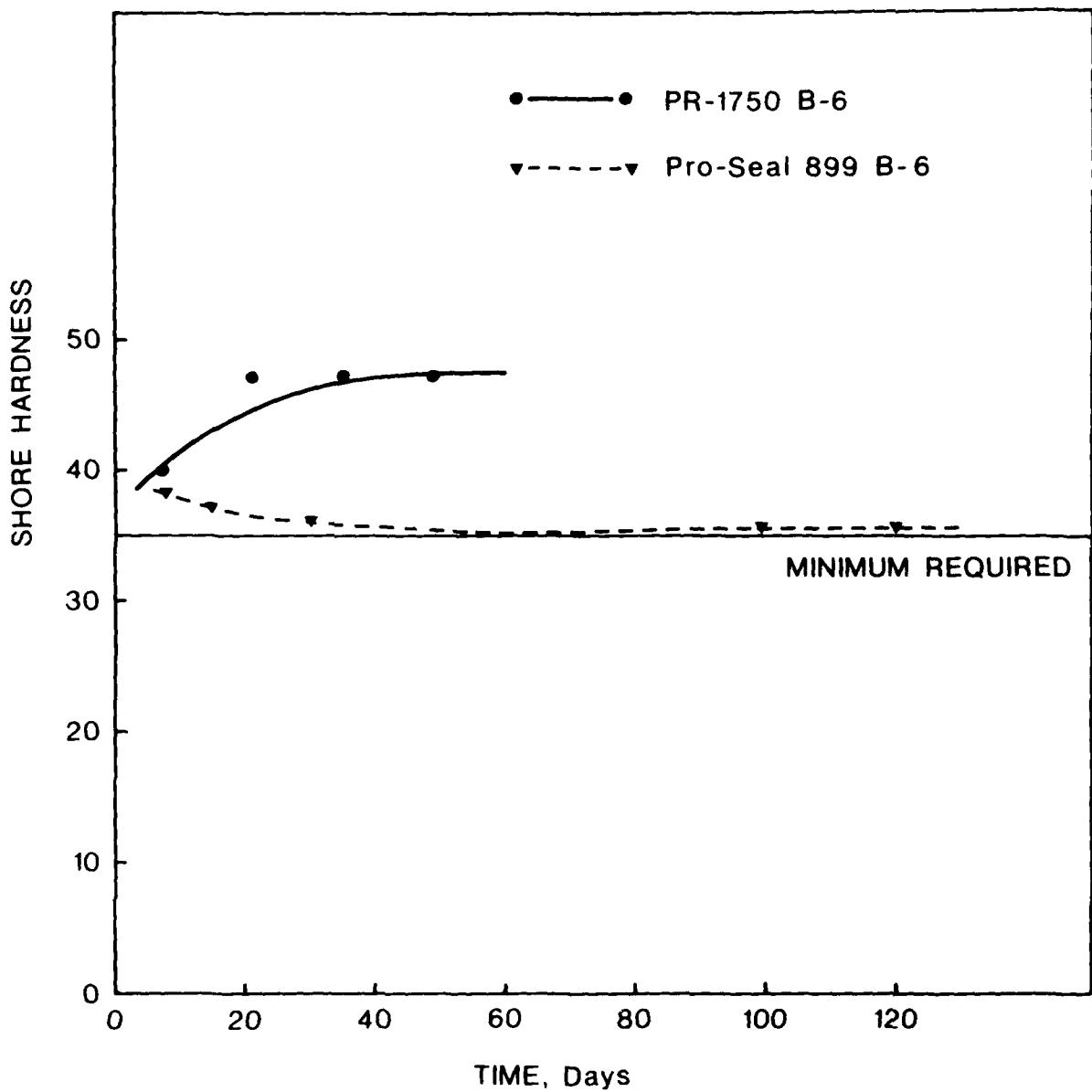


FIG. 2 - Variation of hardness with storage time at -20° to -40°C for class B-6 sealants. (Similar hardness measurements were obtained at all temperatures and the averaged results are shown above).

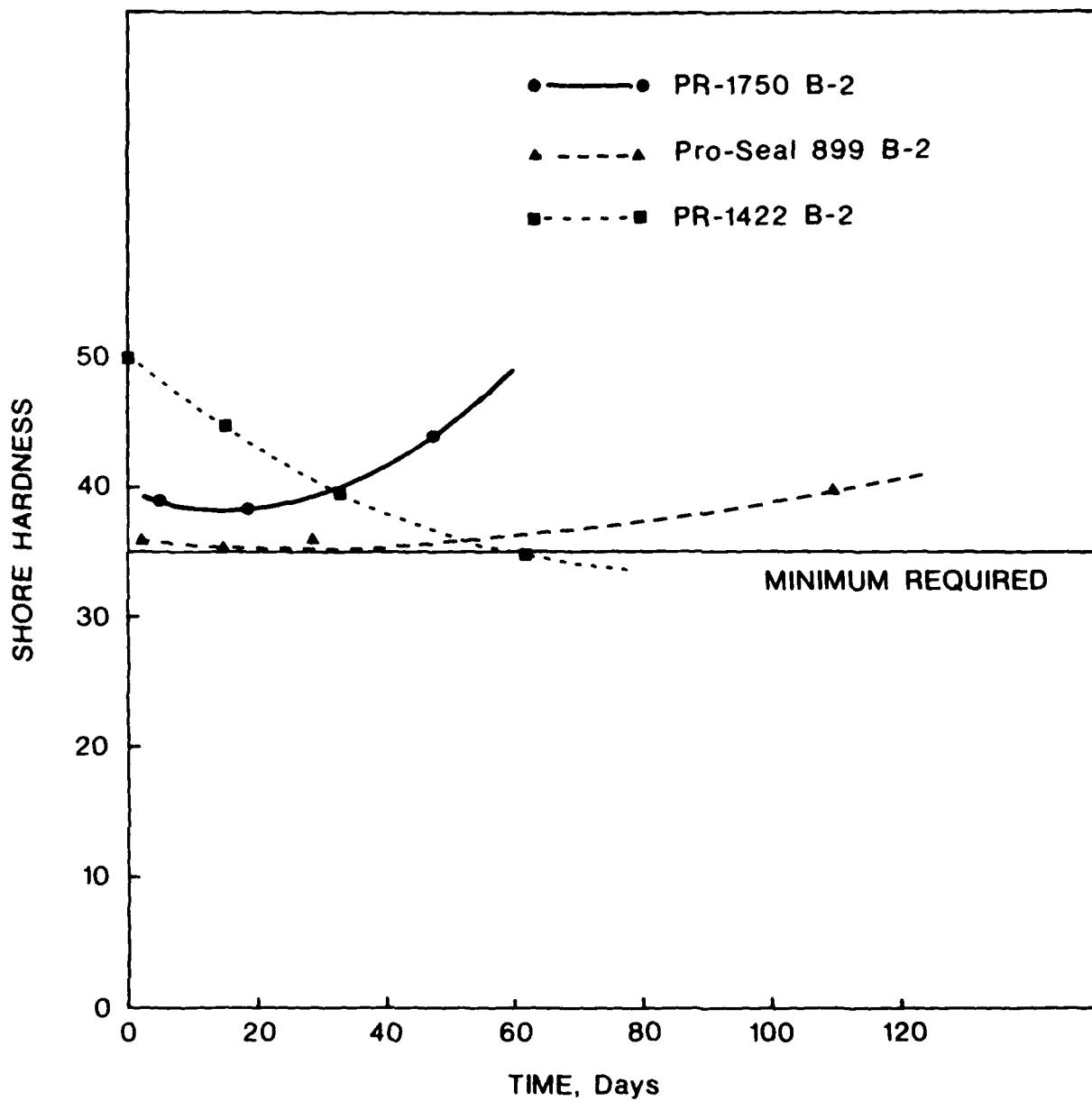


FIG. 3 - Variation of hardness with storage time at -20° to -40°C for class B-2 sealants. (Similar hardness measurements were obtained at all temperatures and the averaged results are shown above).

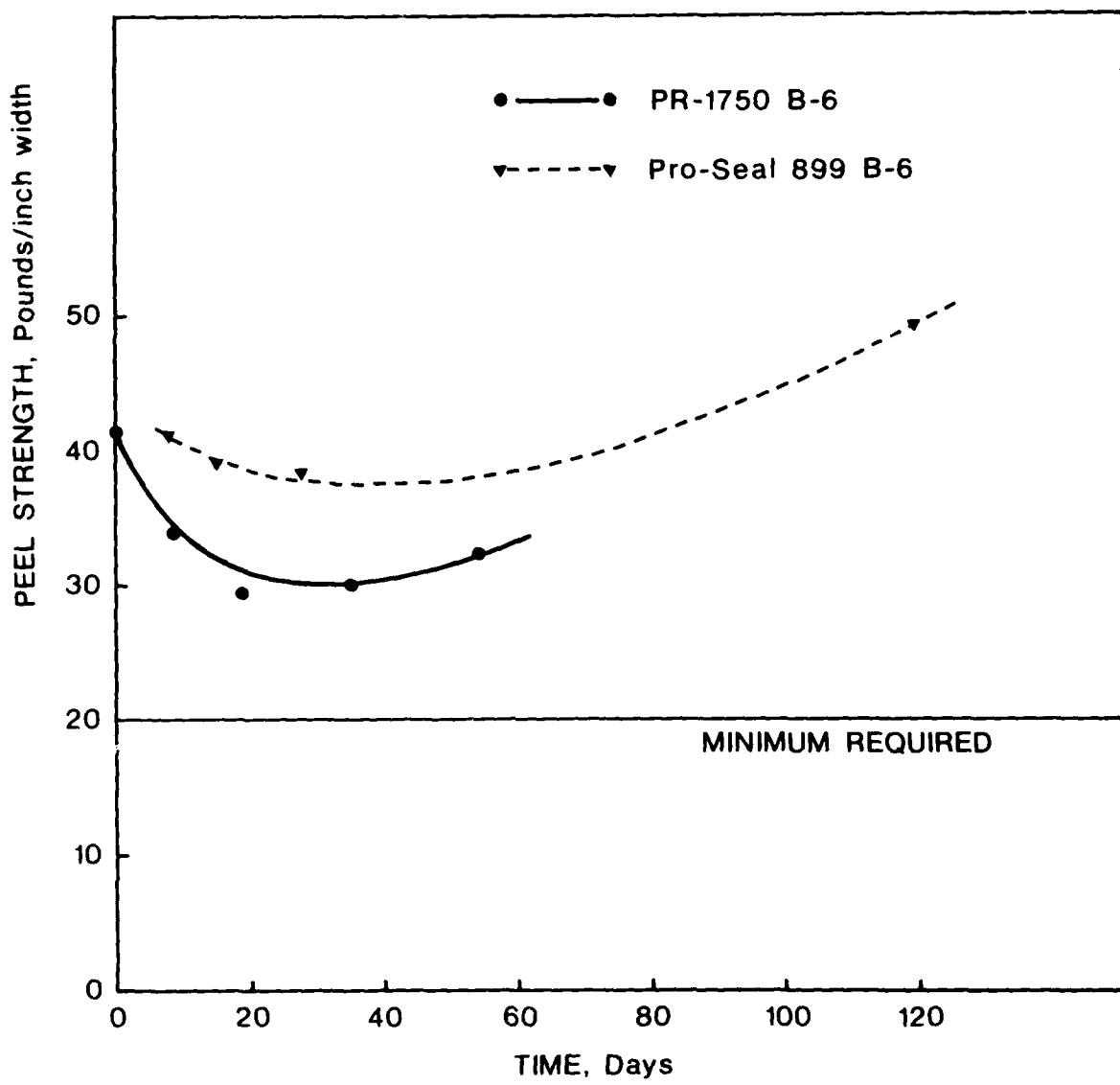


FIG. 4 - Variation of peel strength with storage time at -20° to -40°C for class B-6 sealants. (Similar peel measurements were obtained at all temperatures and the averaged results are shown above).

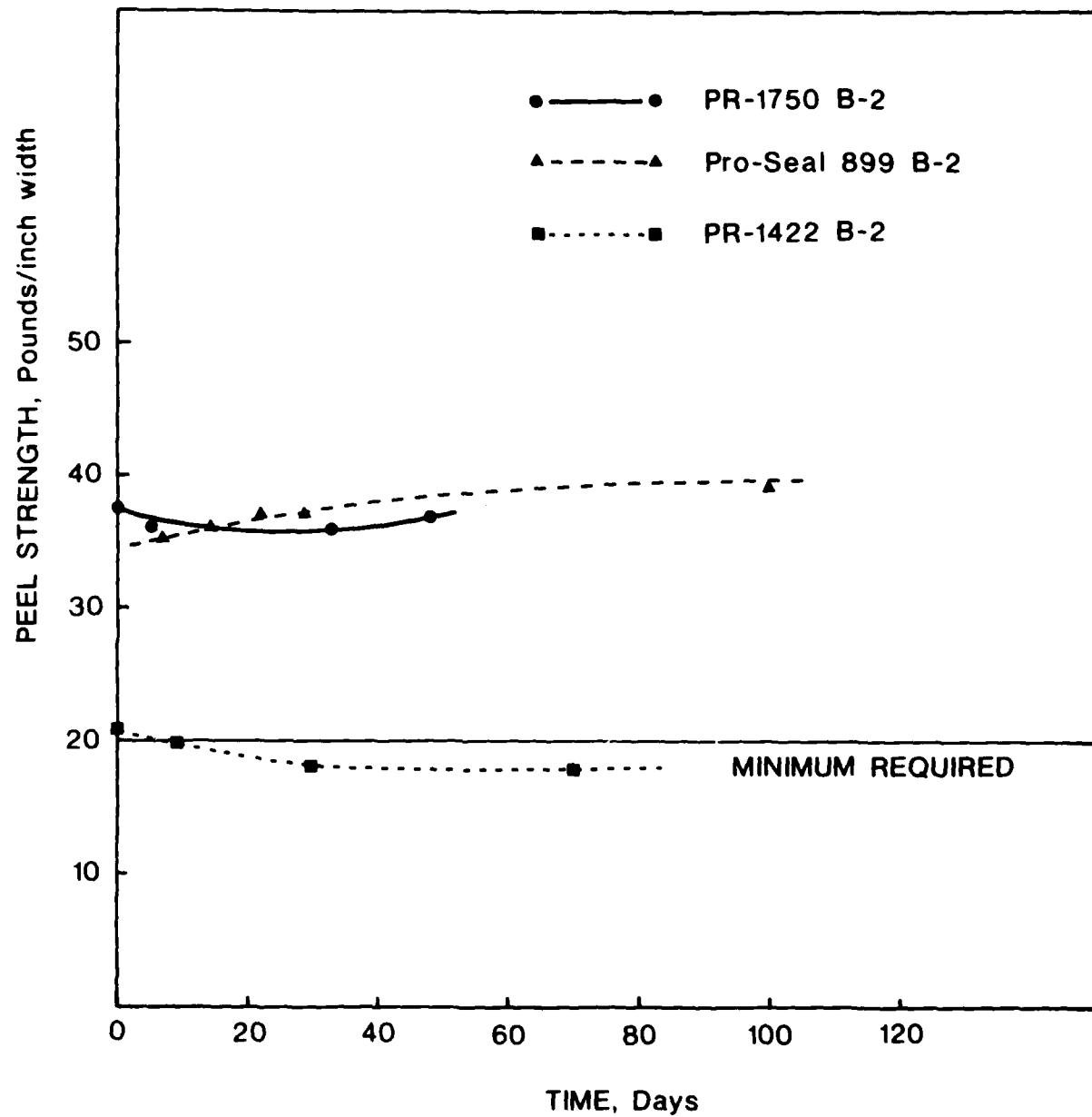


FIG. 5 - Variation of peel strength with storage time at -20° to -40°C for class B-2 sealants. (Similar peel measurements were obtained at all temperatures and the averaged results are shown above).

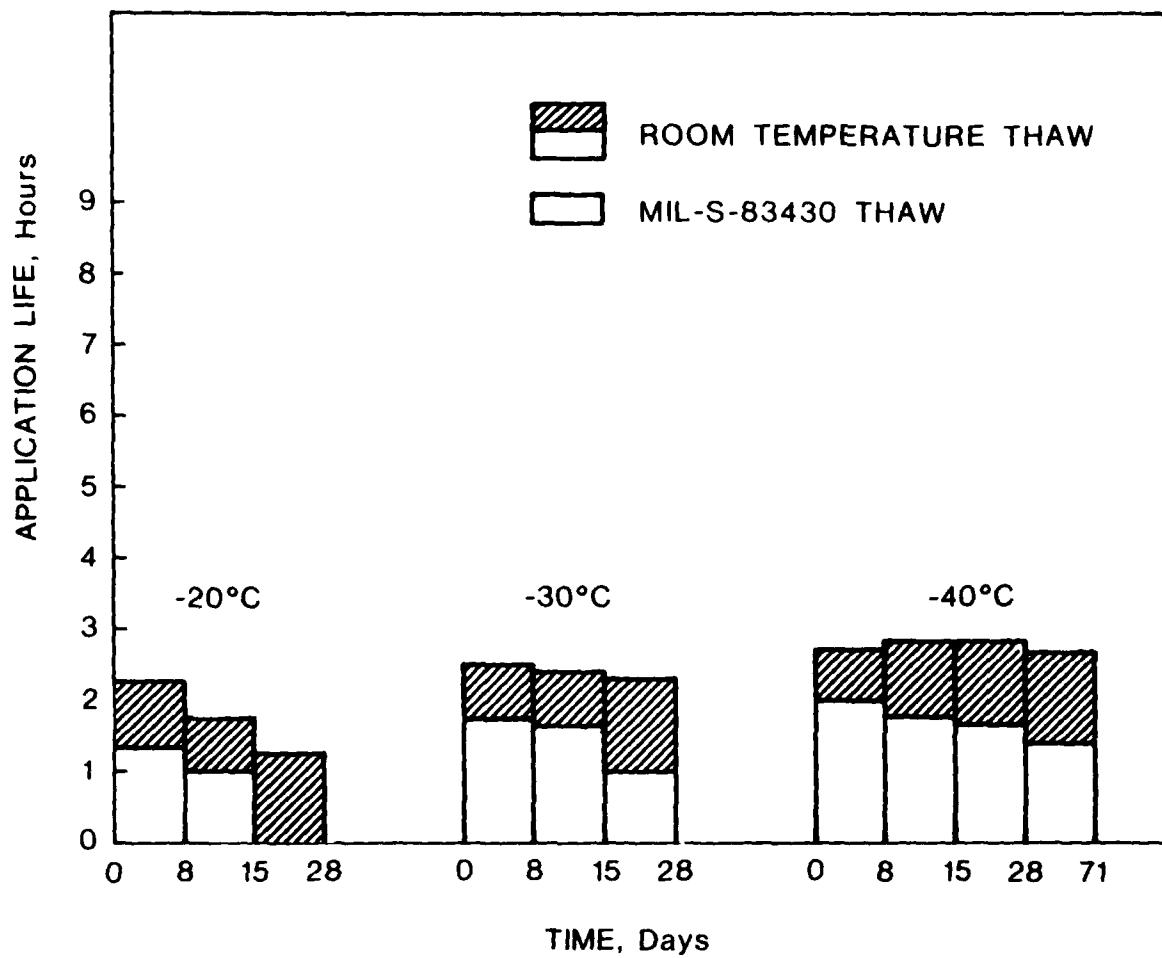


FIG. 6a - Application life of PR-1422 B-2 sealant stored at low temperatures (-20° to -40°C).

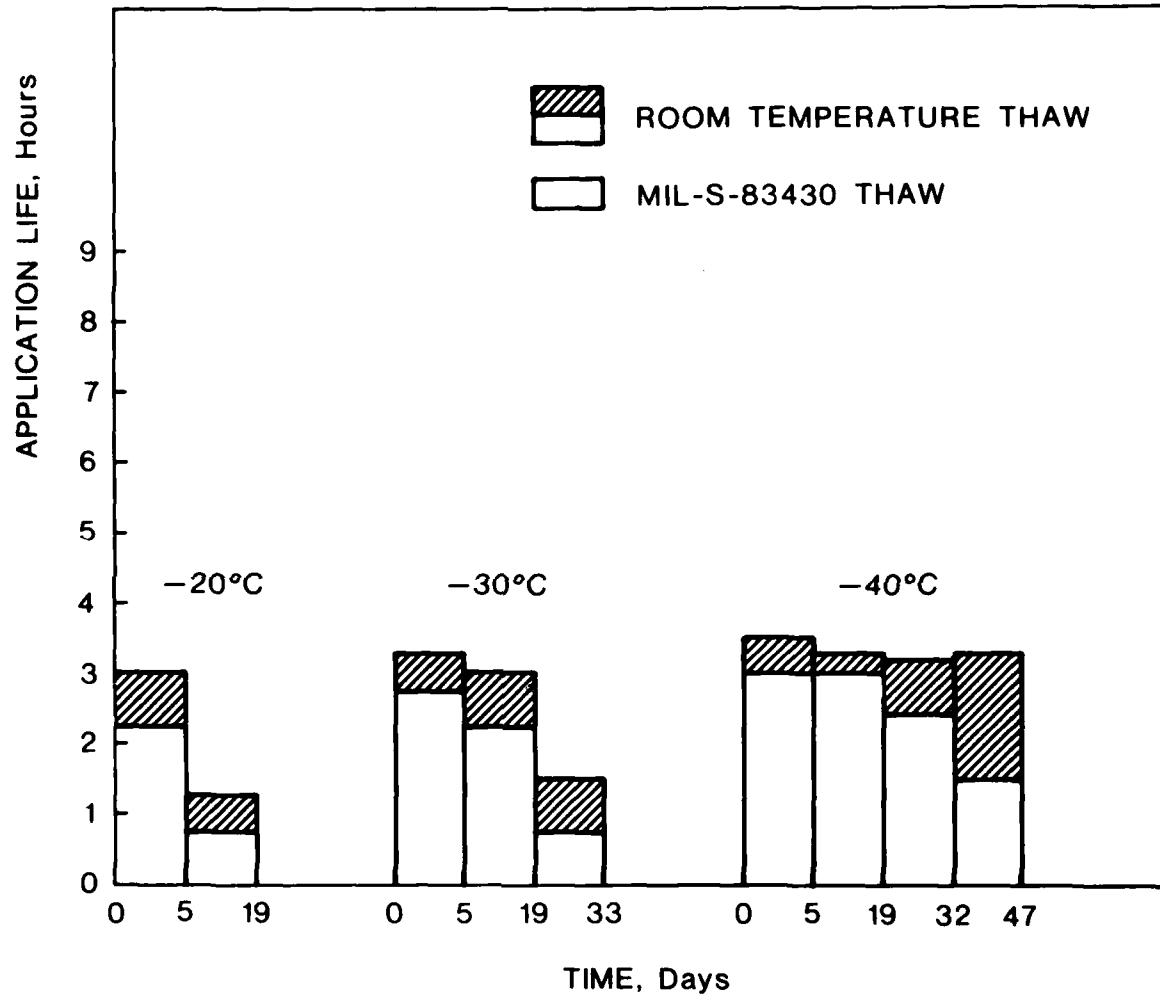


FIG. 6b - Application life of PR-1750 B-2 sealant stored at low temperatures (-20° to -40°C).

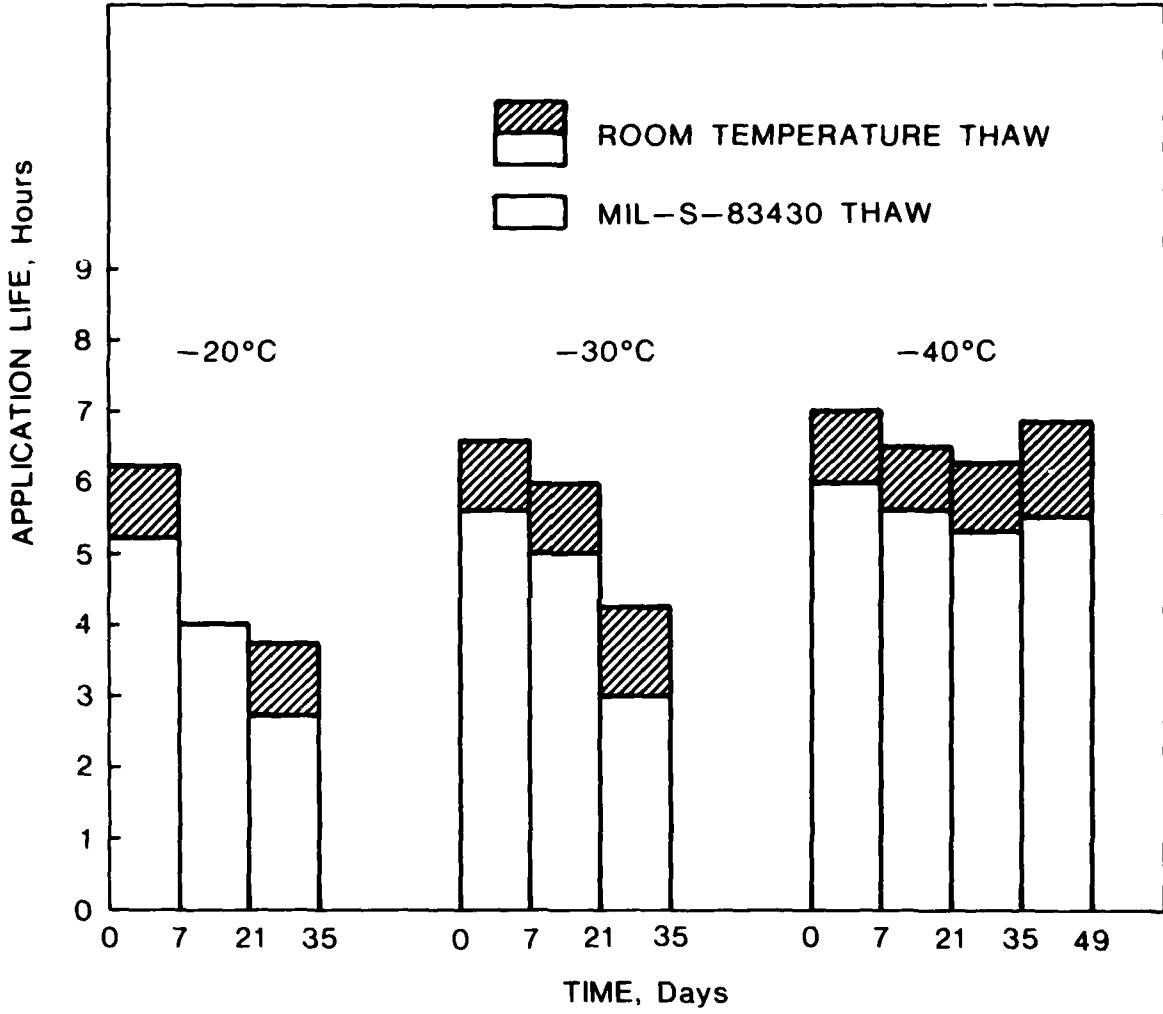


FIG. 6c - Application life of PR-1750 B-6 sealant stored at low temperatures (-20° to -40°C).

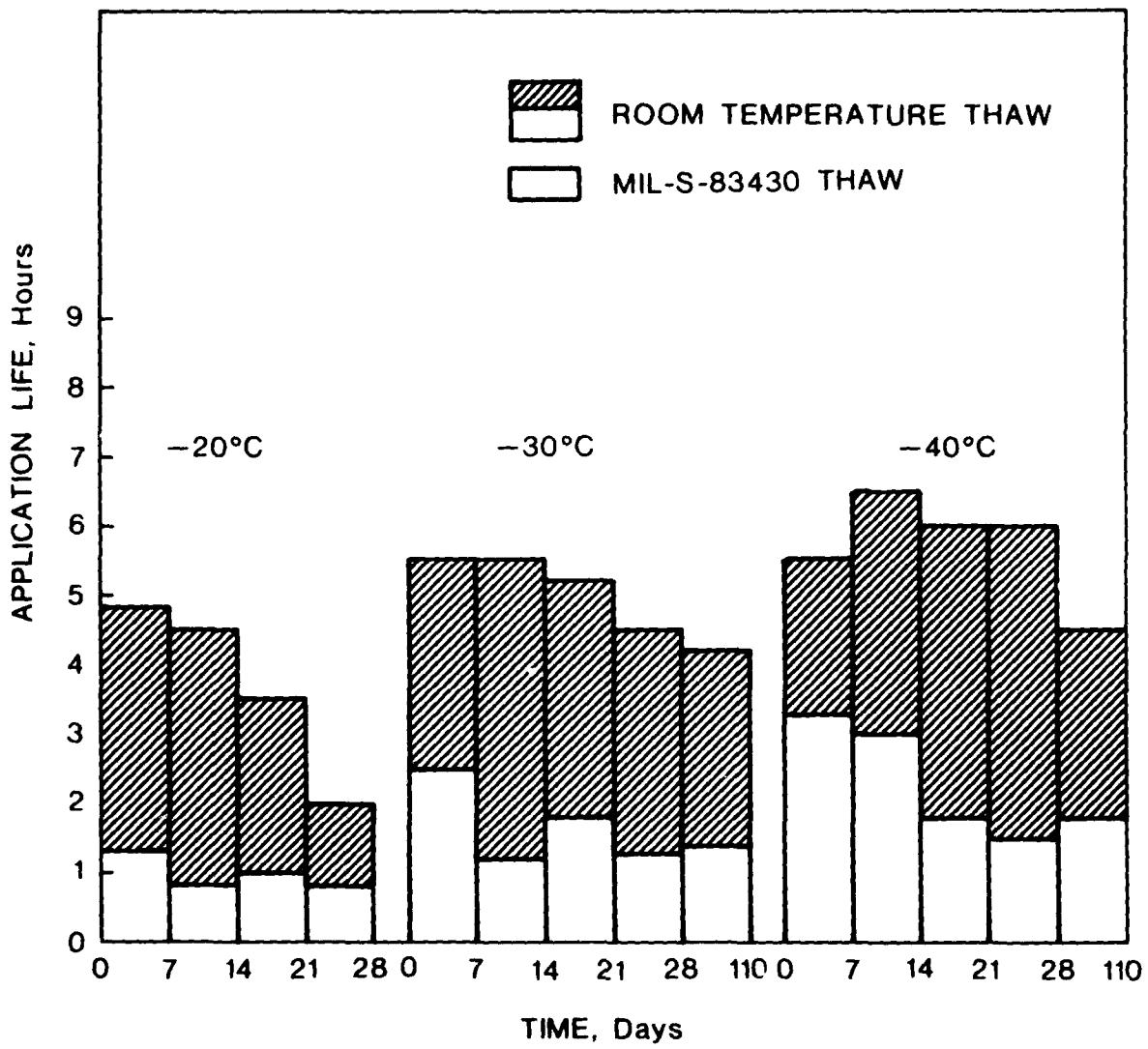


FIG. 6d - Application life of Pro-Seal 899 B-2 sealant stored at low temperatures (-20° to -40°C).

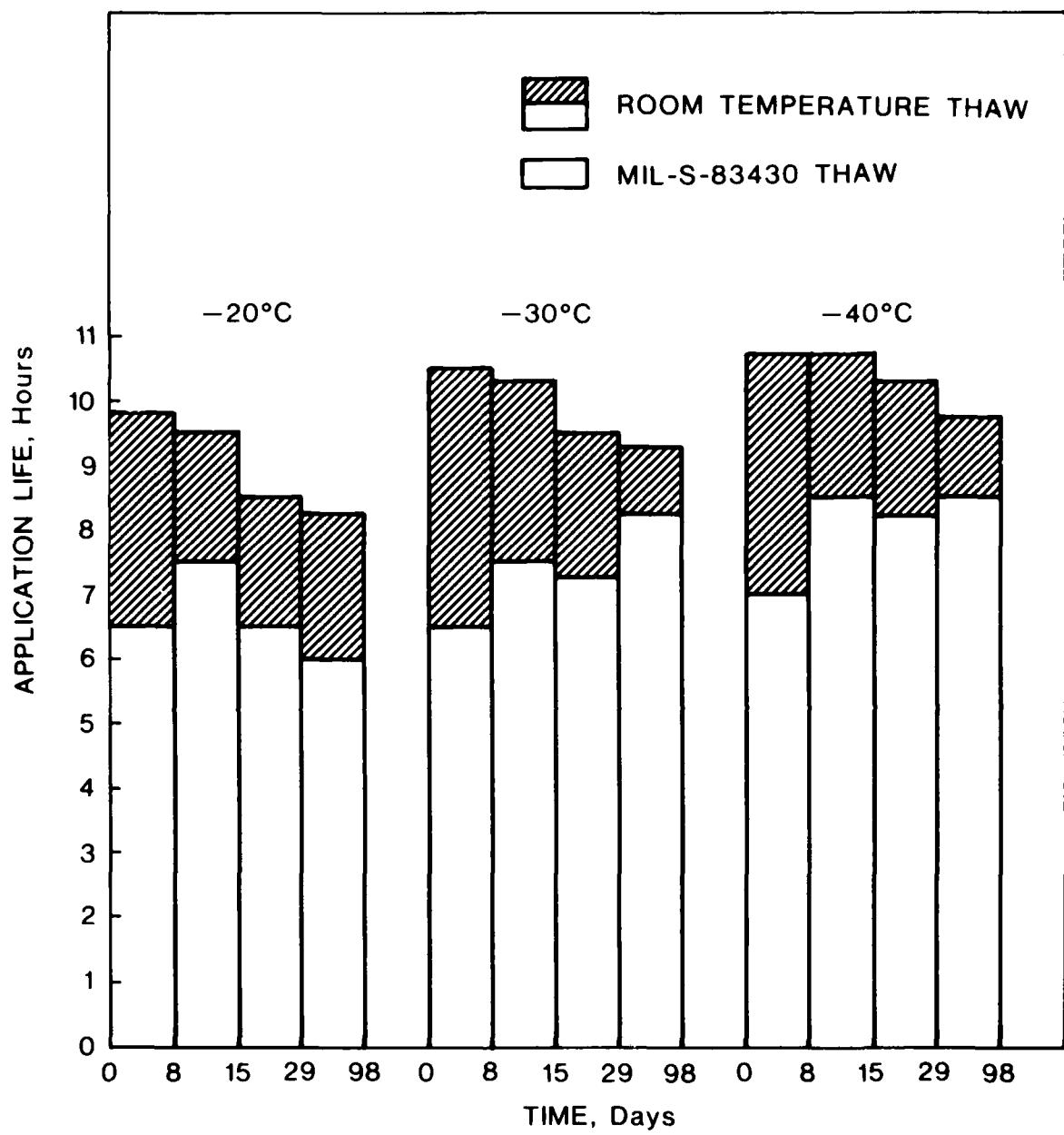


FIG. 6e - Application life of Pro-Seal 899 B-6 sealant stored at low temperatures (-20° to -40°C).

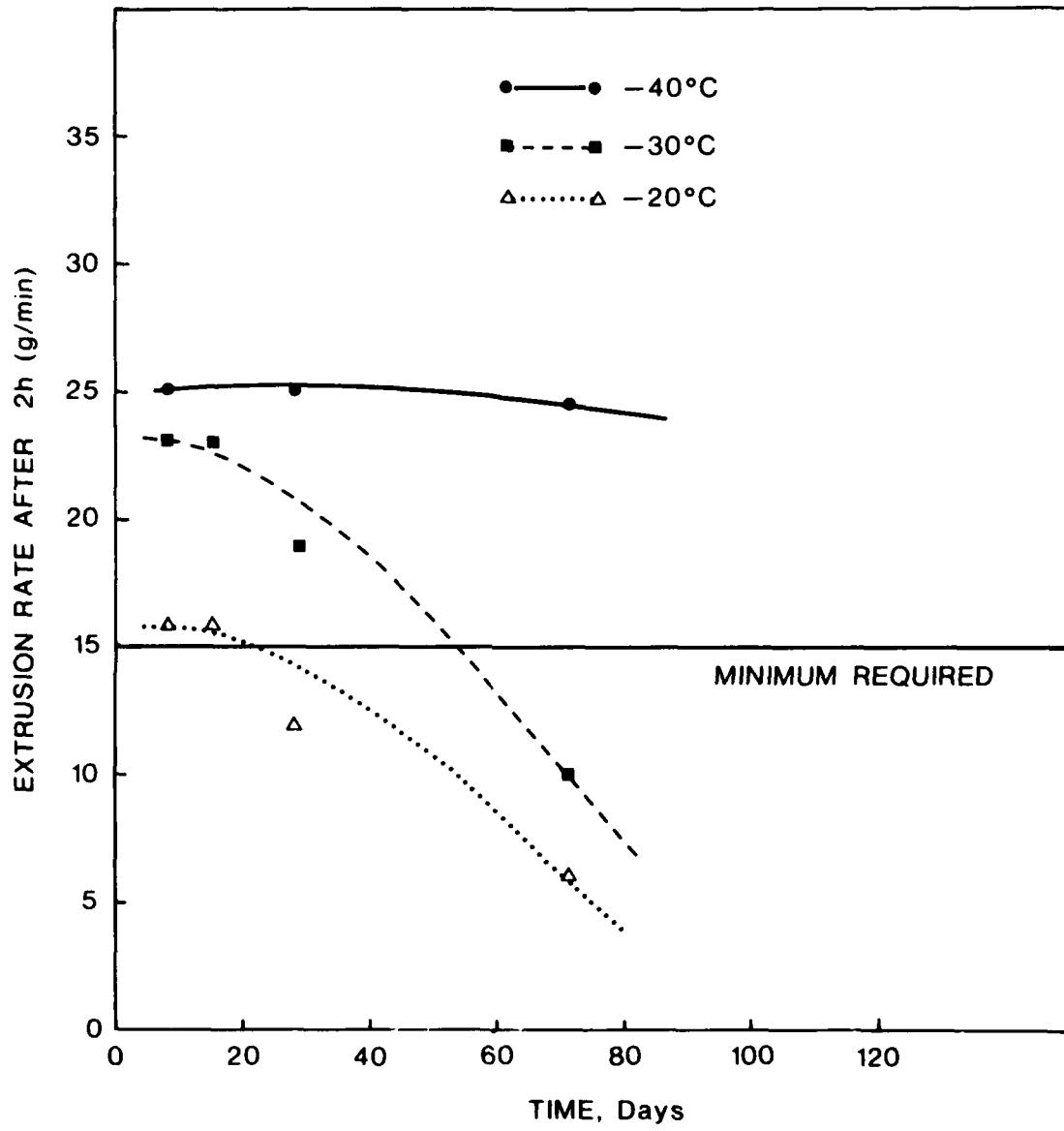


FIG. 7 - Variation in extrusion rate with storage time for PR-1422 B-2 sealant at -20°C to -40°C (room temperature thaw).

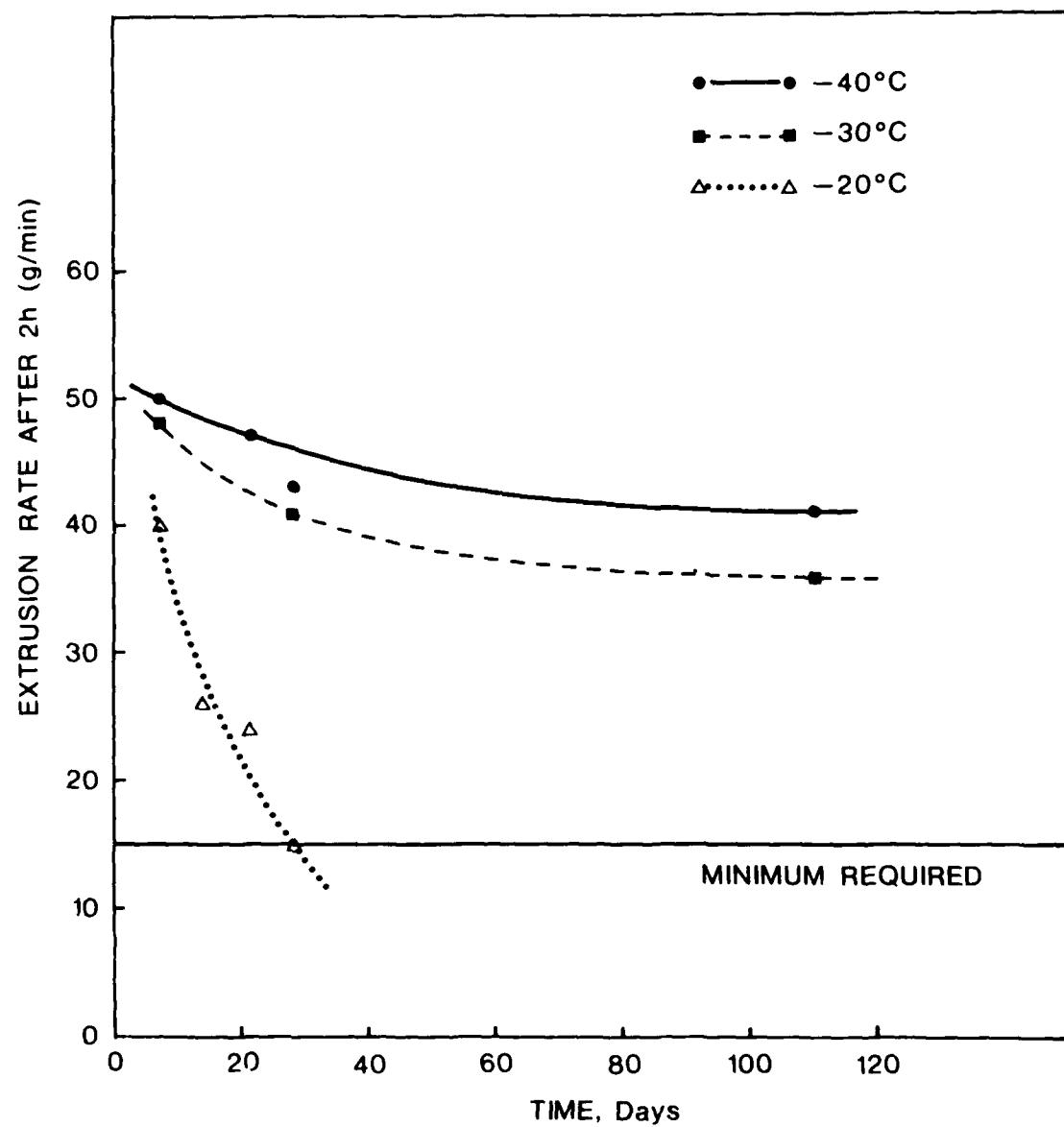


FIG. 8 - Variation in extrusion rate with storage time for Pro-Seal 899 B-2 sealant at -20°C to -40°C (room temperature thaw).

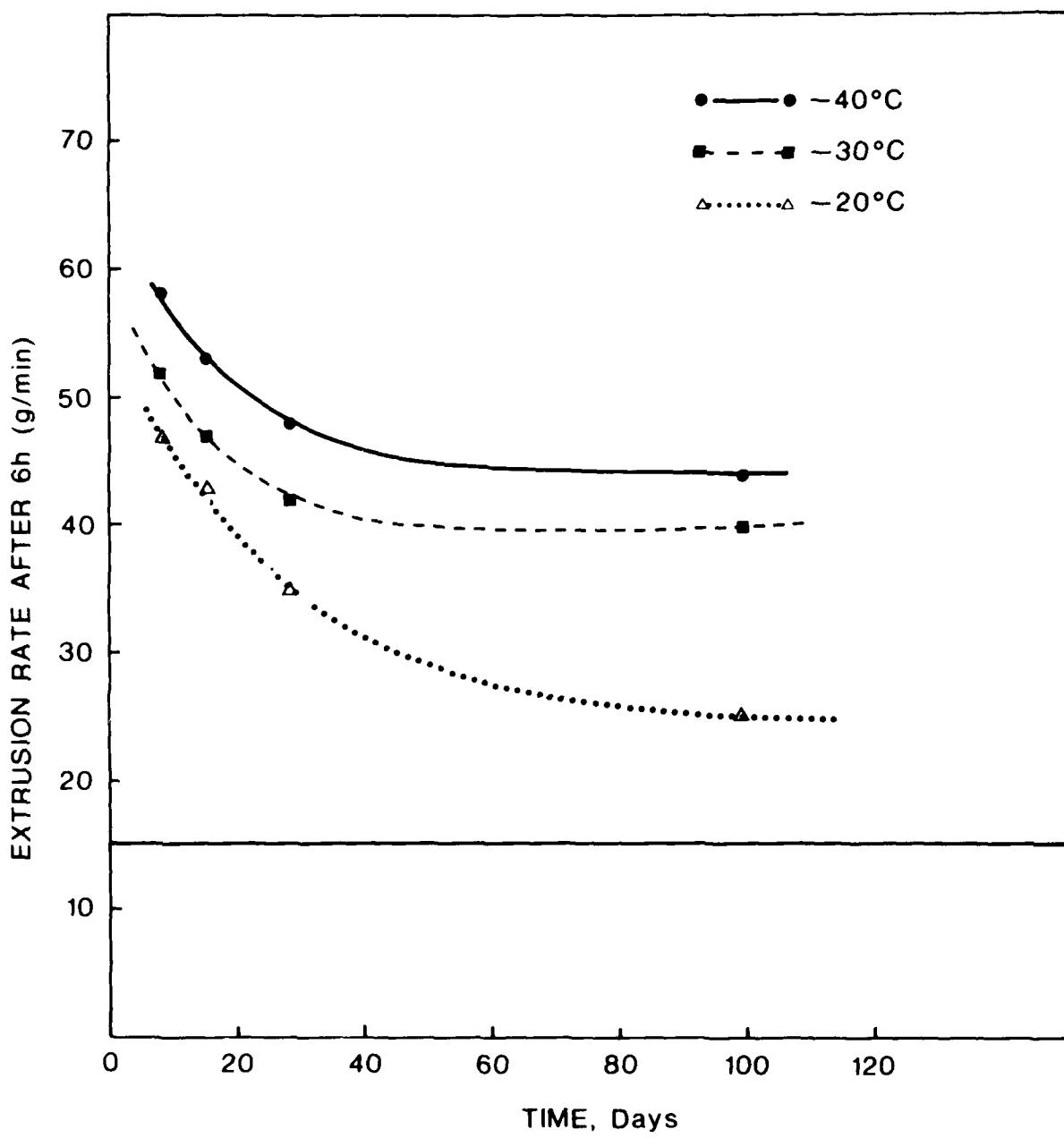


FIG. 9 - Variation in extrusion rate with storage time for Pro-Seal 899 B-6 sealant at -20°C to -40°C (room temperature thaw).

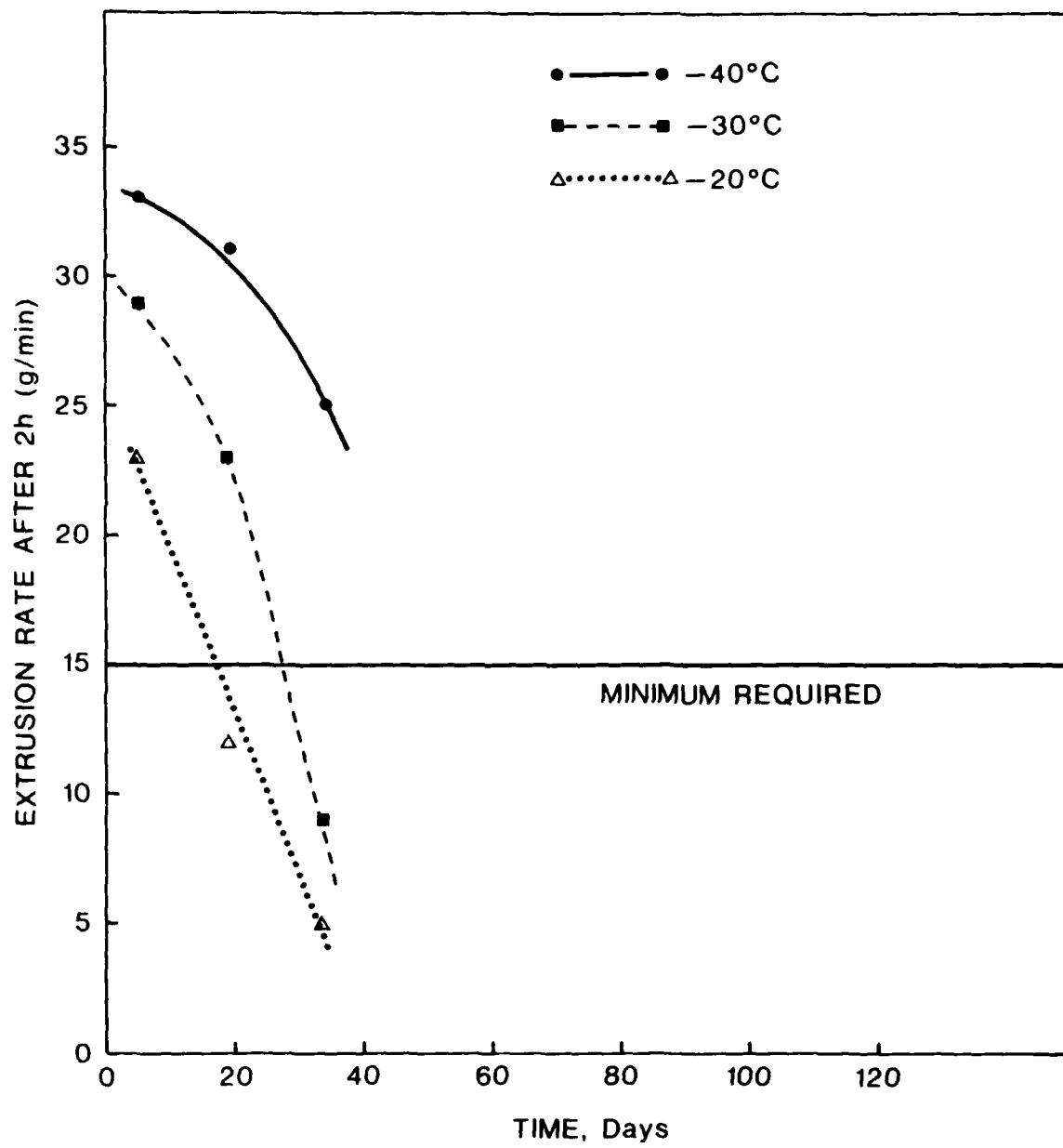


FIG. 10 - Variation in extrusion rate with storage time for PR-1750 B-2 sealant at -20°C to -40°C (room temperature thaw).

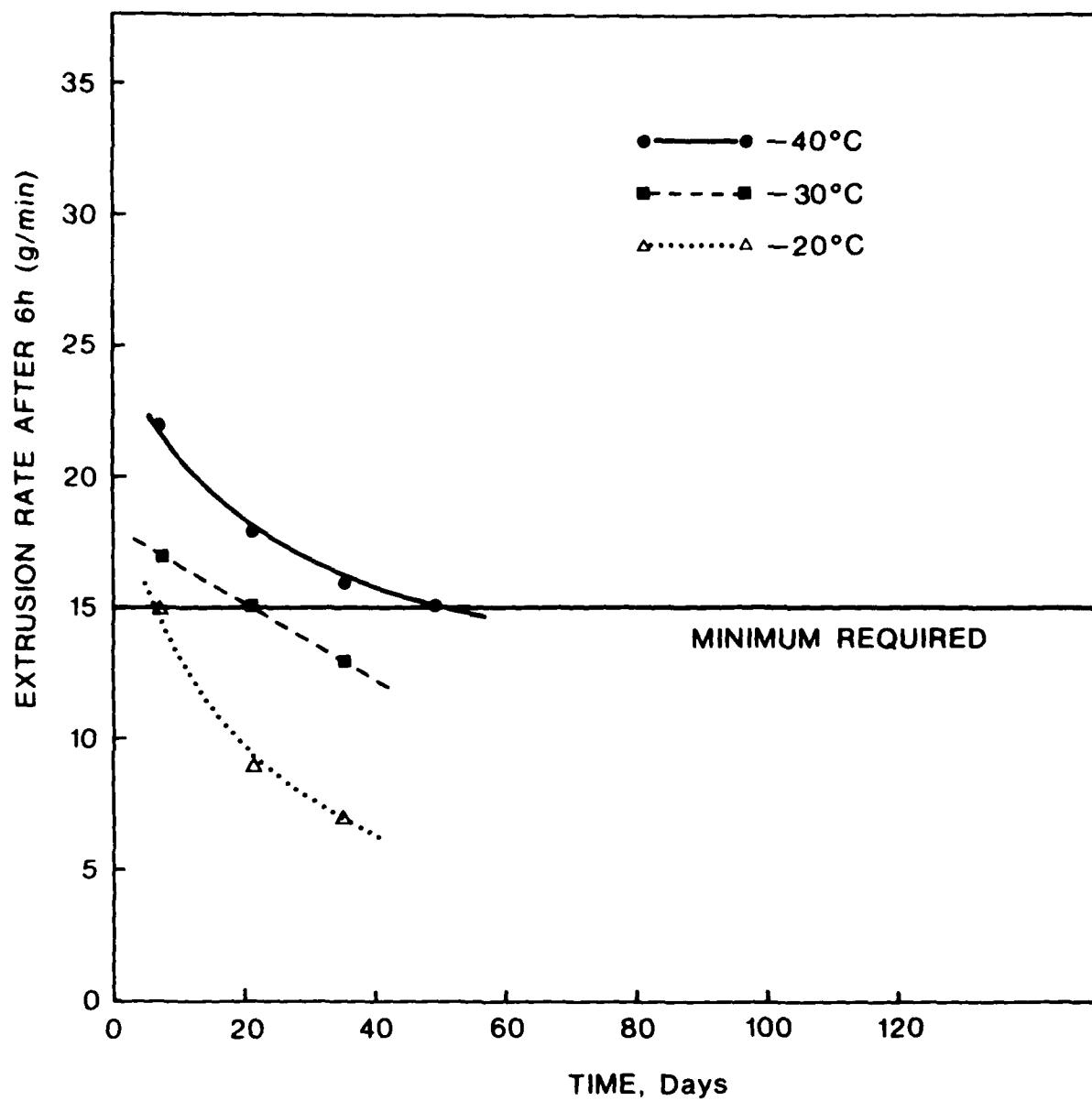


FIG. 11 - Variation in extrusion rate with storage time for PR-1750 B-6 sealant at -20°C to -40°C (room temperature thaw).

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